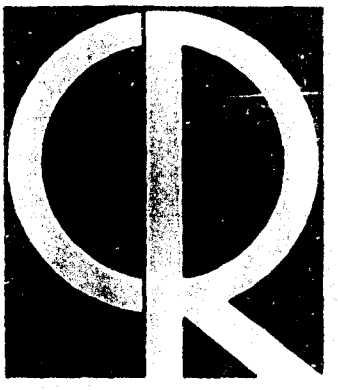
AD 444676

Instrumentation Papers
No. 41



Improved Closed-System Evaporation Crystallizer

JOHN L. SAMPSON
MARY A. DiPIETRO

AFCRL-64-418
MAY 1964

Instrumentation Papers
No. 41

Improved Closed-System Evaporation Crystallizer

JOHN L. SAMPSON
MARY A. DiPIETRO

Reprinted from THE REVIEW OF SCIENTIFIC INSTRUMENTS, Vol. 34, No. 10, October 1963

SOLID STATE SCIENCES LABORATORY PROJECT 5621

AIR FORCE CAMBRIDGE RESEARCH LABORATORIES, OFFICE OF AEROSPACE RESEARCH, UNITED STATES AIR FORCE, L.G. HANSCOM FIELD, MASS.

Improved Closed-System Evaporation Crystallizer

JOHN L. SAMPSON AND MARY A. DIPIETRO

Air Force Cambridge Research Laboratories, Office of Aerospace Research, Bedford, Massachusetts

(Received 31 May 1963; and in final form, 25 June 1963)

AN improved closed-system evaporation crystallizer employing a concentration gradient for growing single crystals has been developed and tested by growing crystals of several materials from solution. The device is based on a Soviet design,[1] the baffle system of which permits some of the unsaturated liquid to bypass the nutrient on the way to the growing zone. The present design has its baffles differently arranged to enhance the transport of the material from the nutrient region to the crystallizing region.

The outer container b of the crystallizer (Fig. 1), which has a tight-fitting dome-shaped top a, is placed in a water bath up to approximately the level of the liquid inside to maintain a temperature above ambient. Solvent evaporates from the surface of the liquid within the crystallizer, condenses on the dome and upper wall of the outer container, and runs down into outer chamber c. This chamber is separated from chamber e by cylinder d, whose top extends above the liquid level, but whose bottom has openings to allow free communication between chambers c and e. Nutrient material h is placed at the bottom of either or both of these chambers. As solvent accumulates in chamber c, hydrostatic pressure causes flow past the nutrient material, some of which dissolves to cause a higher concentration in chamber e than in chamber c. Cylinder f, closed at the bottom and with its top below the liquid level, allows liquid to pass into inner chamber g while keeping solid nutrient out. A seed k consisting of a small single crystal is suspended in chamber g at approximately the top of cylinder f. The liquid in the vicinity of the seed is agitated by a magnetic stirring rod i, externally driven.

Although chambers c and e communicate, the more nearly saturated liquid in e is usually denser than the relatively unsaturated liquid in c, and the liquid level in c is often noticeably higher. In the case of sodium chloride a difference of $\frac{7}{8}$ in. in a 6-in. height (chamber c) was noted. This is in close agreement with the height difference expected on the basis of pure water in c and saturated solution of specific gravity 1.2 in e, and indicates that the rate of flow of solvent is great enough to overcome diffusion effects, which would tend to equalize the concentration throughout the crystallizer.

Reprinted from
THE REVIEW OF SCIENTIFIC INSTRUMENTS
Vo. 34, No. 10 October 1963

Several models of this device have been developed, differing in dimensions, materials, and details of construction. One type uses a hot plate and insulation jacket rather than a water bath, and another type employs direct stirring in lieu of the externally driven magnet. Although laboratory-scale models of 4 to 6 in. o.d. have been used throughout these experiments, a larger apparatus of 12-in. diameter is now undergoing tests.

Thus far the following crystals have been grown from aqueous solution: aluminum ammonium sulfate, nickel ammonium sulfate, sodium silicofluoride, barium nitrate, barium silicofluoride, and silver sulfate. Small crystals of sodium chloride have also been grown from an undoped aqueous solution and from a methanol solution. Seeds of several of these materials have been grown from the stock chemicals by dropping a few grains of powder into the saturated solution in chamber g.

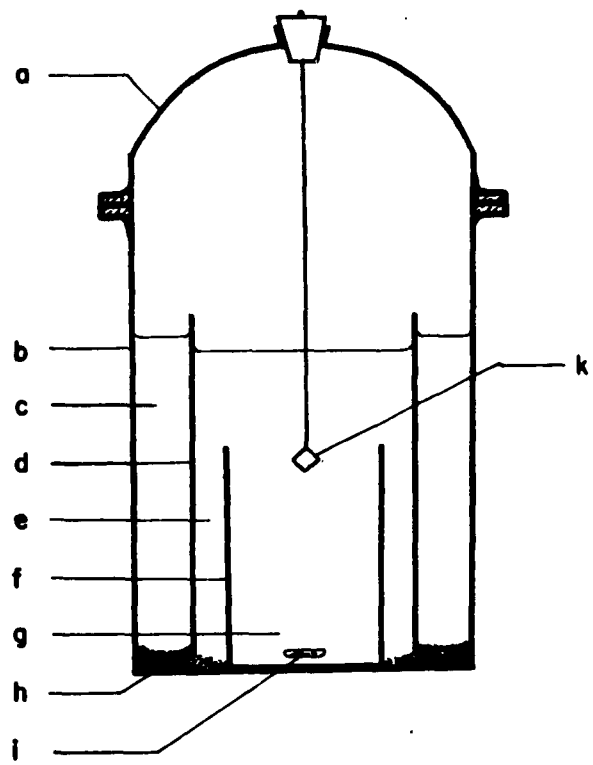


FIG. 1. Diagram of the crystallizer.

The closed system, in which the solvent is not lost but recirculates, allows the crystallizer to be operated for long periods with a minimum of attention. The device, therefore, shows promise for growing crystals of materials of low solubility. No studies have been made as yet of the purity or dislocation density of the crystals grown in this device. Research is continuing on optimum temperature and stirring rate.

The authors wish to thank Dennis Priesner, Wentworth Institute, for his assistance and helpful discussion.

[1] A. G. Karpenko, L. M. Belyaev, B. V. Vitovskii, and G. F. Dobrzhanskii, Kristallografiya 6, 146 (1961) [translation: Soviet Phys.—Cryst. 6, 120 (1961)].

INSTRUMENTATION PAPERS
(Formerly: Instrumentation for Geophysics and Astrophysics)

No. 1. A Digital Electronic Data Recording System for Pulse-Time Telemetering, *Gilbert O. Hall, Feb 1953.*
No. 2. A Rocket-Borne Equipment for the Measurement of Infrared Radiation, *R. M. Slavin, Feb 1953.*
No. 3. Balloon-Borne Conductivity Meter, *S. C. Coroniti, A. Nazarek, C. S. Stergis, D. E. Kotas, D. W. Seymour and J. V. Werme, Dec 1954.*
No. 4. Magnetic Compensation of Aircraft, *J. McClay and B. Schuman, Aug 1955.*
No. 5. Lovotron-A Low Voltage Triggered Gap Switch, *E. H. Cullington, W. G. Chace and R. L. Morgan, Sep 1955.*
No. 6. Balloon-Borne Air Sampling Device, *Charles W. Chagnon, Apr 1957.*
No. 7. Instrumentation for Studies of the Exploding Wire Phenomenon, *W. G. Chace and E. H. Cullington, Aug 1957.*
No. 8. Device for Lowering Loads from High-Altitude Balloons, *W. C. Wagner and F. X. Doherty, Jul 1958.*
No. 9. Equipment and Techniques for In-Flight Deployment of Long Train Instrumentation Packages from High Altitude Balloons, *Arlo E. Gilpatrick and Romain C. Fruge, Sep 1958.*
No. 10. Study of a Phosphor Light Pulser, *Michael R. Zatzick, Sep 1960.*
No. 11. Study of a Pulsed Logarithmic Photometer, *Michael R. Zatzick, Sep 1960.*
No. 12. Theoretical Analysis of the PAR-Scope: An Oscilloscope Display for Weather Radars, *Edwin Kessler, III, Jul 1959.*
No. 13. Evaluation of Visual Distance Computer, CP-384(XD-1), *P. I. Hershberg, Apr 1960.*
No. 14. Hypsometer for Constant Level Balloon, *W. C. Wagner, Jun 1960.*
No. 15. Magnetic Photomultiplier with Large Cathode for Extreme Ultraviolet, *L. A. Hall and H. E. Hinteregger, Dec 1960.*
No. 16. AIDE -- Altitude Integrating Device, Electronic, *P. I. Hershberg, J. R. Griffin and R. H. Guenthner, Dec 1960.*
No. 17. Switching Devices for Very High Currents, *E. H. Cullington and W. G. Chace, April 1961.*
No. 18. Cedar Hill Meteorological Research Facility, *D. W. Stevens, Jun 1961.*
No. 19. Evaluation of Control Monitor AN/GGA 11 Prototype, *R. S. Menchel, Aug 1961.*
No. 20. A Microwave Refractometer with Fast Response and Absolute Digital Recording, *R. H. Shaw and R. M. Cunningham, Mar 1962.*
No. 21. Superpressure Balloon for Constant Level Flight, *L. Gruss, Jul 1962.*
No. 22. Measurement Range Required of Meteorological Equipment, *A. Court and H. Salmela, Aug 1962.*
No. 23. Proportional Counter Spectrometer for Solar X-Rays Between 1 and 10 Angstroms, *L. Heroux, J. E. Manson, and R. Smith, Dec 1962.*
No. 24. A Simplified Sonic Anemometer for Measuring the Vertical Component of Wind Velocity, *J. Chandran Kaimal, Jan 63.*
No. 25. Evaluation of Modification to Antenna of Rawin Set AN/GMD-2, *Konstantin Pocs, Jan 63.*
No. 26. Evolution of the Design of the Anna I Satellite Optical Beacon, *T. Wirtanen, Feb 63.*
No. 27. Seismic Model Impactor, *K. C. Thomson and J. A. Hill, Aug 63.*
No. 28. Error Analysis of the Modified Humidity-Temperature Measuring Set AN/TMQ-11, *R. W. Lenhard, Jr., Major, USAF, and B. D. Weiss, Aug 63.*
No. 29. Evaluation of a Varacter Diode Parametric Amplifier for Rawin Set AN/GMD-2, *Konstantin Pocs, Sep 1963.*
No. 30. The Hydromagnetic Wave Tube, *R. E. Murphy and M. H. Bruce, Sep 1963.*
No. 31. A Time-of-Flight Mass Spectrometer Adapted for Studying Charge Transfer, Ion Dissociation, and Photoionization, *W. Hunt, Jr., et al., Sep 1963.*
No. 32. Moire Fringe Measuring System for Far Infrared Interferometric Spectrometer, *E. V. Loewenstein, Sep 1963.*
No. 33. Opposed Anvil Basic Design Considerations, *R. E. Riecker, 1/Lt USAF, Dec 1963.*
No. 34. New Shear Apparatus for Temperatures of 1000°C and Pressures of 50 kb, *R. E. Riecker, 1/Lt USAF, K. E. Seifert, 1/Lt USAF, Nov 1963.*
No. 35. Accuracy of Meteorological Data Obtained by Tracking the ROBIN With MPS-19 Radar, *Robert W. Lenhard, Jr., Major, USAF, Margaret P. Doody, Dec 1963.*
No. 36. An Application of Induction Heating to Rock Deformation Apparatus, *R. E. Riecker, 1/Lt, USAF, Mar 1964.*
No. 37. A Monopole Phased-Array Feed for Spherical Reflectors, *W. C. Mavroides, L. S. Dorr, April 1964.*
No. 38. Equipment and Techniques for Low Temperature Electron Irradiations, *L.F. Lowe, C. Jimenez, and E.A. Burke, April 1964 (REPRINT).*
No. 39. Lunar Thermal Emission Measurements and Related Antenna Considerations, *John P. Castelli, April 1964 (REPRINT).*
No. 40. Continuous Zone-Refining Apparatus, *John K. Kennedy, April 1964 (REPRINT).*

No. 41. Improved Closed-System Evaporation Crystallizer, *John L. Sampson and Mary A. DiPietro, May 1964 (REPRINT).*

No. 42. Calibration of a Flyable Mass Spectrometer for N and O Atom Sensitivity, *R.S. Narcisi, H.I. Schiff, J.E. Morgan and H.A. Cohen, June 1964 (REPRINT).*